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SINTERING METHOD AND DEVICE

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a method and device for sintering a material in a short period of time by directly applying pressure and current as a result of relatively moving the sintering subject material and heating area while restricting the heating area of the sintering subject material, such as metal and ceramics, to a specific position. The present invention provides a manufacturing method and device suitable for obtaining a sintered body in the form of a long rod or having an uneven cross section.

2. Description of the Related Art

According to the pressurized sintering method employing a direct current, since it is possible to raise the temperature of the sintering subject material at high speed, the manufacturing time can be significantly shortened in comparison to the conventional sintering method employing atmosphere heating.

Generally, the conventional heat sintering method pursuant to a direct current adopts a method of disposing and pressurizing electrodes for current heating at both ends in the axial direction of the sintering subject and simultaneously applying heat thereto (for example, c.f. Japanese Patent Laid-Open Publication No. 2000-239707).

Nevertheless, when heating with such direct current, since the heating value at the contact site of the two in a current path will become considerably larger in comparison to the portions of other sintering subject powders, a thermal gradient will occur from the electrode contact surface toward the

center of the sintering material (position that is distant from the electrode).

Therefore, when manufacturing a sintered product having a long current path such as a rod-shaped material, there is a problem in that it is extremely difficult to sinter the overall material at an even temperature.

Further, with a material in which the cross section of the sintered body is uneven in the lengthwise direction in relation to the current path (i.e., materials with irregular section profiles), since the electrical resistance will change pursuant to the area difference of the cross section perpendicular to the current path, there is a problem in that the heating value will change and an even sintered body cannot be obtained.

Therefore, with the conventional pressurized sintering method employing a direct current, there is a problem in that it is difficult to manufacture a product having an even material quality from a rod-shaped material of a certain length or longer and a stepped material which has an uneven cross section.

Accordingly, a proposal has been made for a method of disposing and heating electrodes at both sides of a sintering subject in substitute for the conventional method of disposing and pressing electrodes for current heating at both ends in the axial direction of the sintering subject (for example, c.f. Japanese Patent Laid-Open Publication No. H10-259405). Nevertheless, here, since the electrodes and sintered body are subject to the processes at a fixed position, it is not possible to continuously sinter a long material.

Moreover, from the perspective of performing continuous sintering, a proposal has been made for sandwiching the sintering subject powder between rolls to be made a thin plate and electrifying and heating it with roll-shaped electrodes (for example, c.f. Japanese Patent Laid-Open Publication No. H9-268302). Nevertheless, this method is limited to the manufacture of thin plates, and there is a problem in that it is not possible to sinter

components of other shapes.

SUMMARY OF THE INVENTION

In view of the foregoing circumstances, an object of the present invention is to provide a sintering method and device that are superior in sintering ability, wherein even if the sintered body is in the form of a long rod or has an uneven cross section, the sintered body is uniform in quality.

As a result of conducting intense study to obtain a sintered body in a rod shape or which has an uneven cross section, the present inventors discovered that the foregoing object can be achieved by restricting (limiting) heating zone in the sintering subject material, and effecting sintering while relatively and successively moving the sintering subject material and current portion.

In other words, based on the foregoing discovery, the present invention provides:

1. A method of performing direct current pressurized sintering to powder in a mold having a cylindrical molding space, wherein sintering is continuously effected while relatively moving a current portion and a sintering subject;
2. A sintering method according to paragraph 1 above, wherein the sintering powder material disposed in the cylindrical mold is pressurized from the end of the mold, an electrode movable in the lengthwise direction of the mold is disposed around the mold, and sintering is effected by energizing and heating the sintering powder material;
3. A sintering method according to paragraph 2 above, wherein the sintering powder material is pressurized from both ends of the mold;
4. A sintering method according to any one of paragraphs 1 to 3 above, wherein an electrode connection terminal assembly affixed to the periphery

of the mold and having a space portion capable of moving freely on a single axis is provided, and sintering is effected by the connection terminal assembly moving the current portion;

5. A sintering method according to paragraph 1 above, wherein fixed electrodes are disposed around a fixed cylindrical die, sintering powder material is filled in the die and subject to current pressurized sintering, the raw material powder is pressed from one side of die, the obtained sintered body is pressed from the opposite side of die, and successive sintering is effected thereby;

6. A sintering method according to any one of paragraphs 1 to 5 above, wherein the sintering powder material is sintered in one direction;

7. A sintering method according to any one of paragraphs 1 to 6 above, wherein a long sintering powder material is sintered;

8. A sintering method according to any one of paragraphs 1 to 7 above, wherein a material with an uneven cross section is sintered while setting the heating area;

9. A sintering device for performing direct current pressurized sintering to powder in a mold having a cylindrical molding space while relatively moving a current portion and a sintering subject, comprising an elevation ram capable of position control and which successively moves the mold and sintering subject;

10. A sintering device according to paragraph 9 above, further comprising a pressurizing ram capable of load control and which pressurizes the sintering powder material disposed in the cylindrical mold from one end of the mold;

11. A sintering device according to paragraph 9 or paragraph 10 above, further comprising an electrode ram which presses the current electrodes disposed around the mold or performs such pressing via the current plate;

12. A sintering device according to any one of paragraphs 9 to 11 above, wherein the sintering powder material is sintered in one direction;

13. A sintering device according to any one of paragraphs 9 to 12 above, wherein a long sintering powder material is sintered; and

14. A sintering device according to any one of paragraphs 9 to 13 above, wherein a material with an uneven cross section is sintered while setting the heating area.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic explanatory diagram showing an example of the device of the present invention to be used in the method of manufacturing a long sintered body by fixing the raw material and moving the electrode;

Fig. 2 is a schematic explanatory diagram showing an example of the device of the present invention to be used in the method of manufacturing a long sintered body by fixing the electrode and die, and moving the sintering powder raw material;

Fig. 3 is a schematic explanatory diagram showing an example of the device of the present invention to be used for manufacturing a long sintered body;

Fig. 4 is an explanatory diagram showing the temperature distribution measurement method of the mold upon performing energization via the electrode connection terminal assembly;

Fig. 5 is a diagram showing the temperature distribution measurement result at the time of performing energization via the electrode connection terminal assembly;

Fig. 6 is a diagram showing the outline of the device to be used in the sintering method employed in Example 2;

Fig. 7 is a cross section of the heating area in the position where the thermocouple 12 exists under the sintering conditions of Example 2;

Fig. 8 is a diagram showing the temperature change of the mold (cylinder) when the current path is separated from the pressurizing axis and the mold is heated thereby;

Fig. 9 is a diagram showing the influence of the sintering temperature on the density of the aluminum sintered product obtained as a result of Example 2;

Fig. 10 is a diagram showing the outline of the method of performing sintering while moving the current path; in other words, the heating area;

Fig. 11 is a cross section schematic showing a sintering example of a stepped component illustrated in Example 4;

Fig. 12 is a cross section schematic showing a sintering example of a component (tapered component) in which the diameter thereof is to be changed;

Fig. 13 is a schematic explanatory diagram showing an example of the device of the present invention to be used in the manufacture of a long sintered body employing a still stage and spacer;

Fig. 14 is a schematic explanatory diagram showing the situation of manufacturing a long sintered body upon employing two spacers in the example illustrated in Fig. 13;

Fig. 15 is a schematic explanatory diagram showing the situation of manufacturing a long sintered body by additionally employing four spacers in the example illustrated in Fig. 14; and

Fig. 16 is a schematic explanatory diagram showing the situation of manufacturing a long sintered body by reversing the partially sintered mold and performing final sintering thereto after the sintering illustrated in Fig. 15.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

With the present invention, instead of integrally and wholly heating the sintering subject material and the mold comprising a sintering space as conventionally, a method has been developed, based on the publicly known current press sintering method, of limiting the current portion to a specific position of the mold, relatively moving the sintering object and the heating portion and continuously effecting sintering in one direction so as to enable the manufacture of a sintered material of a rod shape or having an uneven cross section in which the sintering quality is favorable.

Fig. 1 shows the outline of a case of fixing the raw material and moving the electrodes, and Fig. 2 shows the outline of a case of fixing the electrodes and moving the raw material.

As shown in Fig. 1, when fixing the raw material and moving the electrodes, foremost, an electrode connection terminal assembly (movable electrode) 1 having a thickness corresponding to the portion to be heated is attached to the outer wall of a cylindrical mold (cylinder) 3 for filling the raw material powder 2, and then disposed such that it can move freely on a single axis (cylindrical mold).

Meanwhile, the filled sintering powder 2 is pressurized from both ends in the mold with a punch 4. Reference numeral 5 indicates a pressurizing disk. In this situation, the powder is energized to the movable electrode 1 while pressurizing the powder, and the electrode 1 is moved while controlling it to become a desired temperature and speed. As a result, a sintered body having a rod shape or an uneven cross section can be obtained.

Meanwhile, as shown in Fig. 2, when moving the sintering raw material 2, a die 6 connected to the electrode 1 is energized so as to control it to become a desired temperature, and then the raw material powder 2 is

pressurized and fed into a pushrod 10.

Here, by disposing a roll 7 having a rotational resistance or the likes of a secondary die (not shown) having a slightly smaller diameter than the energizing die 6 at the exit side, the raw material powder is pressurized while taking measures to generate a resistance against the progress of the sintered product 9.

According to the foregoing method, a sintered product in the form of a rod shape or having an uneven cross section in which the uniformity of the sintered product is superior can be obtained.

Further, the device according to the present invention, as shown in Fig. 3, may adopt a constitution of comprising a mold 23 having a cylindrical molding space for filling the sintering subject powder 29, disposing punches 24, 25 having an outer shape that is the same size as the inner diameter of the molding space of such mold 23 at both ends (upper and lower ends) of the mold 23, and pressing the sintering subject powder 29 in the mold with this punch 24.

Although the lower punch 25 is usually fixed, and is constituted to press (apply a load to) the sintering subject powder 29 in the mold with the upper punch 24, a constitution of moving the lower punch 25 may also be adopted. The upper punch 24 is pressurized with the pressurizing ram 21. As shown in Fig. 3, a constitution of performing pressurization with the pressurizing ram 21 via the pressurizing disk 33 may also be adopted.

The lower punch 25 is supported with an elevation ram 22 via a movable elevation stage 34. The elevation stage 34 is constituted to support the mold 23 having a cylindrical molding space, and adjusts the height of the mold 23 having a molding space with the elevation thereof.

The electrode 28 for energizing and heating the sintering subject powder in the mold 23 is designed such that it is able to move in the

horizontal direction. This is required to avoid complicating the mechanism of the energization device from the power source.

Further, provided is an electrode pressurizing ram 30 for pressing the current portion of the electrode 28 to the mold. As shown in Fig. 1, a pair of electrode pressurizing rams 30 is provided to the left and right sides. The electrode 28 may be constituted to press against the mold 23 via the current plate 26.

This current plate 26 has a width that corresponds to the heating area 27 of the raw material powder 29 to be sintered. When performing energization directly with the electrode 28 without the current plate 26, the electrode 28 delinates the foregoing heating zone (area).

In Fig. 3, although the current plate 26 is constituted to sandwich the mold from the left and right sides, as a substitute therefor, an energization ring capable of freely moving in the lengthwise direction of the mold 23 and which attaches to the mold 23 may also be used. Here, the width also corresponds to the heating area of the raw material powder 29 to be sintered.

In the foregoing device, powder 29 is filled in the mold 23 having a cylindrical molding space, the elevation stage 34 is temporarily fixed to adjust the height thereof, and the raw material sintering powder 29 is thereafter pressed with the upper punch 24 for pressurizing the mold 23 from the upper end thereof.

Meanwhile, the upper and lower position of the current electrodes 28 is set to match the position of the heating portion of the raw material sintering powder 29, and energization is simultaneously commenced. Current sintering is conducted in a short period of time. When sintering a long rod-shaped material, the adjustment of the stage position may be conducted in steps or continuously. Further, the stage position may be adjusted while performing energization, or upon performing energization intermittently.

In other words, sintering may be performed by pressing the raw material sintering powder 29 with the punch 24 for pressurizing the mold 23 from one end thereof by arbitrarily adjusting the stage position in steps or continuously, and while simultaneously performing energization, or upon performing energization intermittently.

As a result, even if it is a long rod material, the sintering subject can be successively moved from the upper end of the mold 23 and successively (continuously) sintered in steps.

Further, as a result of adjusting the current for energizing the electrode 28 and the load of the pressurizing ram 21 in conjunction with the position of the freely settable stage elevation ram 22, an arbitrary position of the long material may be sintered at an arbitrary temperature while controlling the pressurizing force.

Moreover, even if the cross section shape (electrical resistance) in the sintering subject material changes, so as long as the heating area is made smaller, the absolute value of the different of the heating value at the respective positions pursuant to the change in shape will be small. Therefore, if the thickness t of the current plate 26 for determining the heating area is made sufficiently thin to an extent of not influencing the quality of sintering, a material having an uneven cross section can be sintered favorably.

As described above, the current value for each portion of the sintering powder raw material 29 can be controlled precisely to match the electrical resistance.

With the present sintering method, the sintering powder material 29 can be sintered in one direction, and, as described above, a long sintering powder material 29 can also be sintered with ease. Further, when a material having an uneven cross section is sintered while setting the heating

area, for instance, a rod-shaped material having a small diameter portion and a large diameter portion; that is, a stepped rod-shaped material can also be sintered with ease.

In other words, the present invention yields a significant characteristic in that it is able to easily sinter a long or irregular rod-shaped material with a relatively simple device constitution.

Examples

The present invention is now explained in detail with reference to the Examples. These Examples are merely illustrative, and the present invention shall in no way be limited thereby. In other words, the present invention shall only be limited by the scope of claim for a patent, and shall include the various modifications other than the Examples of this invention.

(Example 1)

As shown in Fig. 4, a graphite electrode connection terminal assembly 11 having a thickness of t = 10.0mm was mounted to a mold filled with aluminum as the raw material powder, and the temperature distribution in relation to the distance from the connection terminal assembly 11 upon energization thereof was measured.

Results of the temperature distribution measured in Example 1 are shown in Fig. 5. It is evident that the temperature becomes lower as the distance from the electrode connection terminal assembly 11becomes longer. From these results, it is clear that the present invention is able to heat only an arbitrary, limited area up to the sintering temperature.

Even when the cross section shape (electrical resistance) in the sintering subject material changes, so as long as the heating area is made smaller, the absolute value of the difference of the heating value at the respective positions pursuant to the change in shape will be small.

Therefore, if the thickness t of the electrode connection terminal assembly 11 for determining the heating area is made sufficiently thin to an extent of not influencing the quality of sintering, a material having an uneven cross section can be sintered favorably.

Further, since heat generation will not occur in an area outside the electrode connection terminal assembly 11, the temperature of this portion will not become higher than the portion covered with the terminal assembly, and the raw material will therefore not overheat or melt.

(Example 2)

As shown in Fig. 6, 3.82g of aluminum having an average grain size of 20μm was filled in a graphite cylinder mold having an outer diameter of ϕ30mm, inner diameter of ϕ15mm, and length of L = 160mm, and this is pressed from the top and bottom with a graphite punch having a length of 80mm.

A graphite electrode connection terminal assembly (perforated square plate) 11 having a ϕ30mm hole in the center and in which the length of one side thereof is 70mm and the thickness is t = 10.0 to 13.2mm was fitted so as to be attached to the side wall of the cylinder. Fig. 7 shows the cross section of the heating portion in which the thermocouple 12 exists.

Here, the material of the electrode connection terminal assembly (perforated square plate) 11 used is the same as the material of the cylinder. As a result, the current will flow to the cylinder, and the raw material powder will be heated with energization when the electrical resistance of the raw material powder in the cylinder is small. Further, when the electrical resistance of the raw material powder is large, the cylinder will generate heat, and the raw material powder will be indirectly heated and sintered.

Incidentally, a method of dividing and attaching the electrode connection terminal assembly 11 to the cylinder, and energizing and heating

the cylinder or the powder in such cylinder may also be employed. There is no particular limitation on the current heating method, and any one of the known methods may be employed.

The electrode 1 was mounted to the terminal board 11 so as to enable energization in the vertical direction with the pressurizing axis. Further, the electrode connection terminal assembly (square plate) was perforated in the center of the mold (cylinder) 3 to form a 7mm hole, and the thermocouple 12 was inserted therein so as to control the temperature and for monitoring.

After the foregoing preparation, pressurization was performed with a load of approximately 10kN while energizing between the electrodes so as to heat the center of the mold (cylinder) to 580°C to 640°C and effect sintering.

Results of the measurement regarding the temperature change of the mold (cylinder) in foregoing Example 2 are shown in Fig. 8. It is evident that the temperature on the opposite side of the sintering subject material is roughly the same as the preset temperature (corresponding to the control thermocouple) shown with the solid line.

In other words, even when energization is effected via the electrode connection terminal assembly 11 fitted in the mold (cylinder), it is possible to control the sintering subject material 2 to become a desired temperature.

Further, results regarding the density of the aluminum sintered product obtained from Example 2 are shown in Fig. 9.

Fig. 9 shows that the density increases pursuant to the rise in the sintering temperature, and the density approximately reached maximum at 640°C. From these results, it is clear that a favorable sintered body can be obtained even upon separating the current path from the pressurizing axis.

Since the terminal assembly connecting the electrodes is merely fitted in the mold (cylinder), it is able to freely move in the lengthwise direction of the cylinder. Therefore, if sintering is effected while moving the terminal

assembly, a long and uniform sintered body can be manufactured so as to implement the present invention.

(Example 3)

As shown in Fig. 10, in accordance with the sintering method depicted in Example 2, 9.54g of aluminum was filled, and a graphite terminal assembly was successively moved from position 1 to position 3 while sintering was effected to the respective positions via current heating.

Upon examining the density of the aluminum sintered product obtained in Example 3, the relative density showed a value of 99% or more. If the distance to be moved is made longer, the manufacture of an even longer product is possible. Thus, it is evident that a rod-shaped sintered product superior in density can be manufactured with this method.

Next, as an example of sintering a material having an uneven cross section while setting the heating area, when considering sintering a stepped component as illustrated in Fig. 11, this case shows the sintering of a stepped sintered product formed from a portion having a large diameter and a portion having a small diameter. Foremost, when sintering the large diameter portion 13, the electrode connection terminal assembly 11 is mounted to the large diameter portion 13 of the raw material powder. Here, the electrode connection terminal assembly 11 is mounted such that it will not cover the raw material powder of the small diameter portion 14.

The current area is the heating area 15 shown with the diagonal line at the upper diagram of Fig. 11. As a result, the powder positioned at the large diameter portion 13 will be heated and sintered.

Next, the electrode connection terminal assembly 11 is moved to the small diameter portion 14, and similarly sintered via current heating. The current heating portion is the center heating area 16 in the lower diagram of Fig. 11. Here, the electrode connection terminal assembly 11 is mounted

such that it will not cover the large diameter portion 13.

At the respective positions, energization matching the electrical resistance can be effected independently. As a result, a stepped component can also be evenly energized and sintered. Incidentally, while the electrode connection terminal assembly 11 is moving, the energization may be stopped, or current for keeping a specific temperature may be flowed. This can be set arbitrarily.

Further, as shown in Fig. 12, even if the material has a tapered portion, if the thickness t of the electrode connection terminal assembly 11 for determining the heating area is made sufficiently thin to an extent of not influencing the quality of sintering, a favorable sintered product can be manufactured.

(Example 4)

As shown in Fig. 13, a sample for sintering testing was prepared by inserting a punch 25 having a length of 10mm to a graphite cylinder mold 23 having an outer size of 40mm square and length of L = 100mm, and in which a hole having a diameter of ϕ15mm was formed therein, such that it will not protrude from the lower end of the cylinder 23, and 26.0g of aluminum powder 29 having an average grain size of 20μm was filled therein.

The height of this sample was adjusted such that the distance from the lower end of the cylinder 23 to the center of the electrode 28 will become 80mm and placed on the stage, and sandwiched with the current plates 26 having a height of 30mm $\times$ width of 40mm mounted to the center of the electrode 28.

A punch 24 having a length of 40mm was mounted on the cylinder 23, and subject to powder compacting at a load of 900kgf. Under these conditions, the electrodes 28 were energized and heated up to 650°C. Incidentally, for temperature control, the thermocouple inserted in the hole

having a depth of 12.0mm formed at the center of the side face at a height of 80mm from the lower end of the cylinder was used.

Next, as shown in Fig. 14, two spacers having a thickness of 10mm were placed on the stage, and, while raising the position of the cylinder 20mm, the position of the thermocouple was lowered 20mm (on the center line of the electrode), and the second heating was effected with the method described above.

Thereafter, this was repeated twice, and a total of four heating steps were conducted to manufacture a rod-shaped sintered product. Incidentally, in the fourth heating step, as shown in Fig. 16, such heating was conducted with the cylinder positioned upside down since it was not possible to insert 60mm worth of spacers on the stage due to the chamber size in the used device.

In this Example, an aluminum sintered body having a length of approximately 55mm was obtained. Upon examining the density of this sintered product, a relative density having a value of 99.7% was obtained.

This result shows that a sufficient numerical value was obtained as the density of the sintered product, and it has been confirmed that the present invention was able to obtain a favorable rod-shaped sintered product. Incidentally, as necessary, as shown in Fig. 15, four additional spacers may be used.

If the scale-up of the device is sought to enable the mold (cylinder) 23 to move even longer, an even longer sintered product can be manufactured by increasing the number of heating steps.

Moreover, if the heating area is made small and the current value is controlled to match the electrical resistance for each portion, sintering can be effect at a fixed temperature even if the cross section shape is changed. Therefore, the present invention enables the sintering and manufacture of a

favorable long product or a component having an uneven cross section.

Incidentally, although only aluminum was used in the Examples, the present invention is not limited to such aluminum material. Powders of other metals or ceramics may also be sufficiently employed.

The present invention proposes a method of sintering while relatively moving the raw material and electrode, and, since it is not necessary to sinter the enter product at once, there is an effect in that the heating area can be made small. Further, since energization is effected through the electrode connection terminal assembly mounted on the mold, heat generation will only occur to the portion corresponding to the thickness of the electrode connection terminal assembly. Therefore, if the thickness of the electrode connection terminal assembly is made thin to a range where the cross section of the sintering subject material will be even, the heat generation of the sintering subject material at such position will become even.

As a result, the temperature variation during sintering can be restrained, and a significant effect is yielded in that a long sintered body or a sintered body having an uneven cross section with superior quality can be manufactured.

Further, as a result of employing the method of sequentially supplying the raw material powder, the continuous manufacture of a rod-shaped material is enabled. As a result, in comparison to the conventional batch production method, a significant effect is yielded in that a considerable improvement in productivity in the sintered material can be expected.